



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of : Confirmation No. 3778  
Kousuke AKIYAMA et al. : Attorney Docket No. 2005\_2076A  
Serial No. 10/562,826 : Examiner Sheeba Ahmed  
Filed December 30, 2005  
OIL-RESISTANT SHEET MATERIAL

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DECLARATION UNDER 37 CFR 1.132

Commissioner for Patents  
P.O.Box 1450  
Alexandria, VA 22313-1450

Sir:

I, Kousuke AKIYAMA, declare as follows:

I am one of the inventors of the above-identified application and am fully familiar with the subject matter and facts set forth therein.

I hold a Bachelor's degree in inorganic chemistry from the Chemistry Department of Science and Engineering of Aoyama Gakuin University in 1994.

From the time of my completion of Graduate School down to the present, I have been employed by Tokushu Paper MFG. Co., Ltd. and have been engaged in research and development in novel and improved paper.

In the last 5 years, I have been engaged especially in oil-resistant sheet, so I am well acquainted with the technical knowledge of Oil-resistant sheet.

I have conducted the following experiments in order to achieve the objects described below.

#### OBJECTS OF EXPERIMENTS

The first object of the following experiments is to show evidence that the combination of polyvinyl alcohol and a fatty acid can produce a coating material with oil resistance and good air permeability characteristics as well, as shown in paragraph [0036] of the present specification.

The second object is to show evidence that the oil resistance of a sheet material can be further improved by using a fatty acid sizing agent instead of other fatty acids such as a fatty acid antifoamer.

#### PREPARATION OF SAMPLES TESTED

A paper substrate having the following composition, a basis weight of which was 42 g/m<sup>2</sup>, was prepared in the same manner as in Example 1 of the present specification.

• Pulp composition

Hardwood bleached kraft pulp	50% by weight
Softwood bleached kraft pulp	50% by weight

• Additives

Epichlorohydrin-based wet strength agent	0.5% by weight
Rosin sizing agent	0.5% by weight
Aluminum sulfate	4% by weight

(Note: Additive rates described above represent weight proportion of the solid content of the additives relative to the solid content of the pulp.)

[Sample 1]

Sample 1 was prepared by applying the coating solution having the following composition to both sides of the paper substrate prepared above so that the total solid of coating layers on both sides by this coating solution was  $3.0 \text{ g/m}^2$ .

(Note: Additive rates described hereinafter represent weight proportion of the solid content relative to the total amount of the coating solution.)

- Carboxyl modified polyvinyl alcohol 5% by weight  
(GOHSENOL T350, product of Nippon Synthetic Chemical Industry Co., Ltd.)
- Crosslinking agent 0.5% by weight  
(WS-570, product of Seiko PMC Corporation)

[Sample 2]

The coating solution having the following composition was used in place of the coating solution of Sample 1.

- Carboxyl modified polyvinyl alcohol 5% by weight  
(GOHSENOL T350)
- Crosslinking agent (WS-570) 0.5% by weight
- Fatty acid sizing agent 0.5% by weight

[Sample 3] (corresponding to Example 3)

The coating solution having the following composition was used in place of the coating solution of Sample 1.

- Hydrophobized starch 5% by weight
- Crosslinking agent (WS-570) 0.5% by weight
- Fatty acid sizing agent 0.5% by weight
- Carboxyl modified polyvinyl alcohol 1.5% by weight  
(GOHSENOL T350)

[Sample 4]

The coating solution having the following composition was used in place of the coating solution of Sample 1.

- Hydrophobized starch 5% by weight
- Crosslinking agent (WS-570) 0.5% by weight
- Carboxyl modified polyvinyl alcohol 1.5% by weight  
(GOHSENOL T350)

[Sample 5]

The paper substrate prepared above (to which no coating solution was applied) was used as Sample 5.

[Sample 6] (corresponding to Example 1)

The coating solution having the following composition was used in place of the coating solution of Sample 1.

- Hydrophobized starch 5% by weight
- Crosslinking agent (WS-570) 0.5% by weight

[Sample 7] (corresponding to Example 2)

The coating solution having the following composition was used in place of the coating solution of Sample 1.

- Hydrophobized starch 8% by weight
- Crosslinking agent (WS-570) 0.8% by weight
- Fatty acid sizing agent 0.8% by weight

[Sample 8]

The coating solution having the following composition was used in place of the coating solution of Sample 1.

- Hydrophobized starch 8% by weight
- Crosslinking agent (WS-570) 0.8% by weight

- Fatty acid antifoamer 0.8% by weight  
(Afranil MG, product of BASF)

[Sample 9]

The coating solution having the following composition was used in place of the coating solution of Sample 1.

- Hydrophobized starch 8% by weight
- Crosslinking agent (WS-570) 0.8% by weight
- Fatty acid 0.8% by weight  
(containing brassidic acid as a main component)

The Summary of the Sample's composition described above is shown in Table 1.

**TABLE 1**

	P V A	Crosslinking agent	Hydrophobized starch	Fatty acid sizing agent	Fatty acid antifoamer	Fatty acid (Brassidic acid)
Sample 1	○	○				
Sample 2	○	○		○		
Sample 3	○	○	○	○		
Sample 4	○	○	○			
Sample 5						
Sample 6		○	○			
Sample 7		○	○	○		
Sample 8		○	○		○	
Sample 9		○	○			○

#### EVALUATION METHOD

##### 1) Air permeability

The air permeability was evaluated by JIS P8117 (1998),

"Paper and Board-Determination of Air Permeance-Gurley Method".

2) Oil resistance (non-heated)

The oil resistance at the time of non-heated was evaluated in the same manner as in paragraphs [0078] and [0079] of the present specification

3) Oil resistance (heated)

The oil resistance at the time of heated was evaluated as follows. First, each Sample was cut into a rectangle having a size of roughly 3cm × 5cm, and a drop of salad oil was put on one surface of each Sample. Next, each Sample was quickly turned over and fixed to the testing stand as shown in Fig. A described below, and then heated for 2 minutes at a temperature of 180°C with the use of the oven. After 2 minutes, the permeation of dropped salad oil to the other side was visually observed.

The evaluation criteria of permeation of salad oil were as follows. The third ranked mark "△" and higher were evaluated as "passing" marks.

◎: Substantially no permeation of salad oil was observed.

○: Permeation of salad oil was observed, but the permeation area was less than or equal to 10% of the drip area of the salad oil.

△: Permeation of salad oil was observed, but the permeation area was less than or equal to 30% of the drip area of the salad oil.

×: Permeation of salad oil was observed, and the permeation area was more than 30% of the drip area of the salad oil.

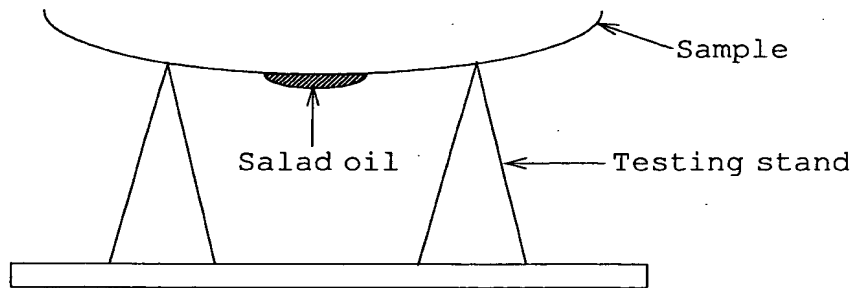


Fig. A

### EVALUATION RESULTS

The evaluation results are shown in Table 2.

**TABLE 2**

	Basis weight (g/m <sup>2</sup> )	Thickness (mm)	Coating amount (DRY) (g/m <sup>2</sup> )	Air permeability (sec.)	Oil resistance	
					heated	non-heated
Sample 1	45.4	0.072	3.5	8100	○	△
Sample 2	44.9	0.074	2.9	1700	×	△
Sample 3	45.2	0.076	3.2	540	◎	◎
Sample 4	45.0	0.071	3.0	3810	△	◎
Sample 5	42.0	0.067	0	80	×	×
Sample 6	45.3	0.072	3.3	2700	×	○
Sample 7	45.3	0.073	3.3	760	◎	◎
Sample 8	45.4	0.073	3.4	680	×	○
Sample 9	45.3	0.072	3.3	590	×	○

### FINDINGS

1) Comparison of Sample 1 with Sample 2 revealed that the

air permeability could be markedly increased by adding a fatty acid sizing agent to a coating layer containing a polyvinyl alcohol and a crosslinking agent. That is, a coating layer by a combination of a polyvinyl alcohol, a crosslinking agent and a fatty acid sizing agent showed superior air permeability to a coating layer consisting only of a polyvinyl alcohol and a crosslinking agent.

2) Comparison of Sample 3 with Sample 4 revealed that the air permeability could be markedly increased by adding a fatty acid sizing agent to a coating layer containing a hydrophobized starch, a crosslinking agent and a polyvinyl alcohol. That is, a coating layer by a combination of a hydrophobized starch, a crosslinking agent, a polyvinyl alcohol and a fatty acid sizing agent showed superior air permeability to a coating layer consisting only of a hydrophobized starch, a crosslinking agent and a polyvinyl alcohol.

3) Comparison of Sample 5 with Sample 1 or 6 revealed that the air permeability was substantially decreased by forming a coating layer consisting of a polyvinyl alcohol and a crosslinking agent or a coating layer consisting of a hydrophobized starch and a crosslinking agent. In addition, comparison of Sample 4 with Sample 6 showed that the air permeability was further decreased by adding a polyvinyl alcohol to a coating layer containing a hydrophobized starch and a crosslinking agent.

4) However, as described in 2), the air permeability could be markedly increased by adding a fatty acid sizing agent to



a coating layer containing a hydrophobized starch, a crosslinking agent and a polyvinyl alcohol.

5) According to the evaluation results of Samples 1 to 4, it can be found that improved oil resistance due to a fatty acid could be obtained only when a coating layer contained a hydrophobized starch. Comparison of Sample 1 with Sample 2 showed that, even though a fatty acid sizing agent was added to a coating layer containing a polyvinyl alcohol and a crosslinking agent, unless the coating layer contained a hydrophobized starch, the oil resistance (heated, non-heated) was not improved, while the air permeability was increased. However, comparison of Sample 3 with Sample 4 revealed that, when a fatty acid sizing agent was added to a coating layer containing a hydrophobized starch, a crosslinking agent and a polyvinyl alcohol, not only the air permeability was increased, but also the oil resistance was improved.

6) Comparison of Sample 7 with Sample 8 revealed that the oil resistance, especially at the time of heated, could be significantly improved by using a fatty acid sizing agent instead of a fatty acid antifoamer.

7) Comparison of Sample 7 with Sample 9 revealed that the oil resistance at the time of heated could be further improved by using a fatty acid sizing agent instead of a fatty acid (containing brassidic acid as a main component).

#### CONCLUSION

As described in findings 1) to 5), it is clear that the

combination of polyvinyl alcohol and a fatty acid can produce a coating material with oil resistance and good air permeability characteristics as well.

As described in findings 6) and 7), it is clear that the oil resistance of a sheet material can be further improved by using a fatty acid sizing agent as a fatty acid.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Dated this 27<sup>th</sup> day of August, 2008

Kousuke Akiyama

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